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5-Chloro-1-(prop-2-ynyl)indoline-2,3-dione

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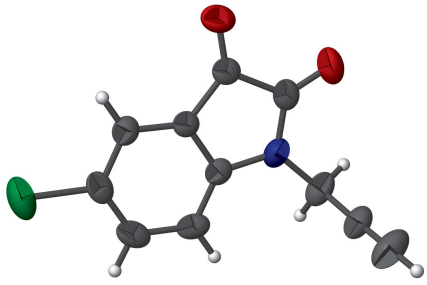
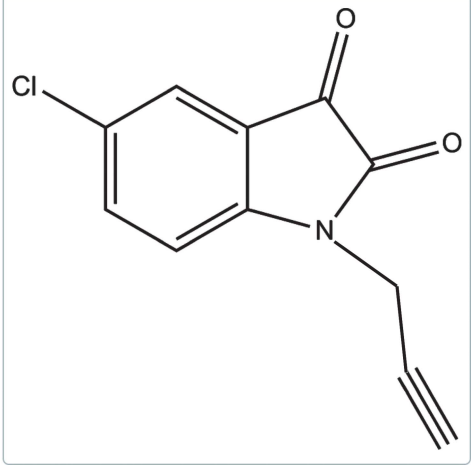
Structural data: full structural data are available from iucrdata.iucr.org

In the title isatin derivative, $C_{11}H_6ClNO_2$, the indoline ring is planar (r.m.s. deviation = 0.009 Å), with the two ketone O atoms lying in the plane and the chlorine atom displaced by 0.036 (1) Å. The dihedral angle between the mean plane of the indoline ring system with that of the propynyl chain is 73 (8)°. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming zigzag chains propagating along the *b*-axis direction. The chains are linked *via* weak π–π interactions [inter-centroid distance = 3.728 (1) Å], forming slabs parallel to the *bc* plane.

3D view



Chemical scheme



Structure description

Isatins (indole-2,3-diones) are used in the synthesis of a large variety of heterocylic compounds (da Silva *et al.*, 2001). Isatin and its derivatives have been reported to show pharmacological actions such as antimicrobial, anticancer, antiviral, anticonvulsant, anti-inflammatory and analgesic (Bhrigu *et al.*, 2010). Biological properties of isatin include a range of actions in the brain and offer protection against certain types of infections (Pandeya *et al.*, 2005). They have been evaluated for antibacterial and antifungal activities (Ramachandran, 2011), and have been reported to possess anti-MES activity (Smitha *et al.*, 2008).

In the title compound, Fig. 1, the indoline ring is planar (r.m.s. deviation = 0.009 Å). Atom Cl1 is displaced from this mean plane by 0.036 (1) Å, while the O atoms, O1 and O2, lie in the plane of the ring system. The indoline ring is nearly perpendicular to the mean plane passing through the 1-propynyl chain as indicated by the C8—N1—C9—C10 torsion angle of 91.0 (2)°.

In the crystal, molecules are linked by C—H···O hydrogen bonds, forming chains along the *b*-axis direction (Table 1 and Fig. 2). The chains are linked by slipped parallel

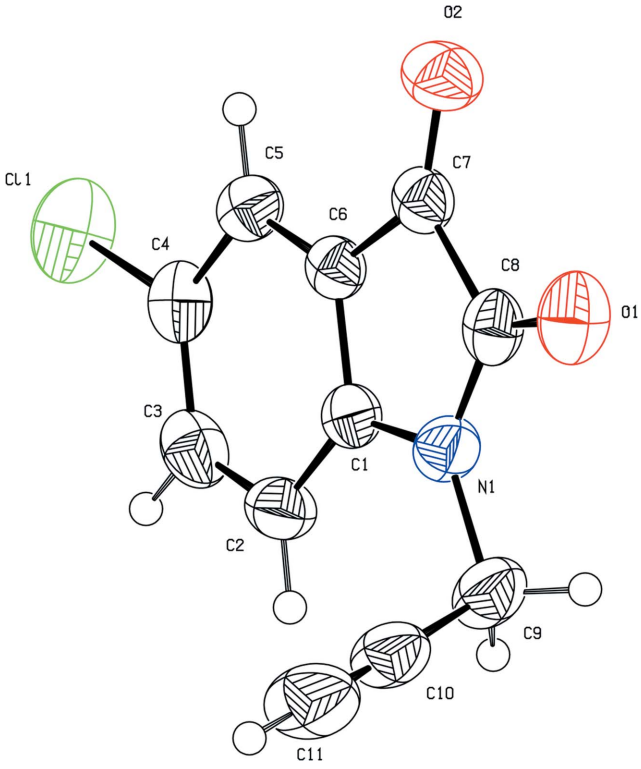


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

π–π interactions, forming slabs lying parallel to the *bc* plane [$Cg1\cdots Cg2^{i}$ = 3.728 (1) Å, inter-planar distance = 3.327 (1) Å, slippage = 1.71 Å, *Cg*1 and *Cg*2 are the centroids of the N1/C1/C6–C8 and C1–C6 rings, respectively; symmetry code: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$].

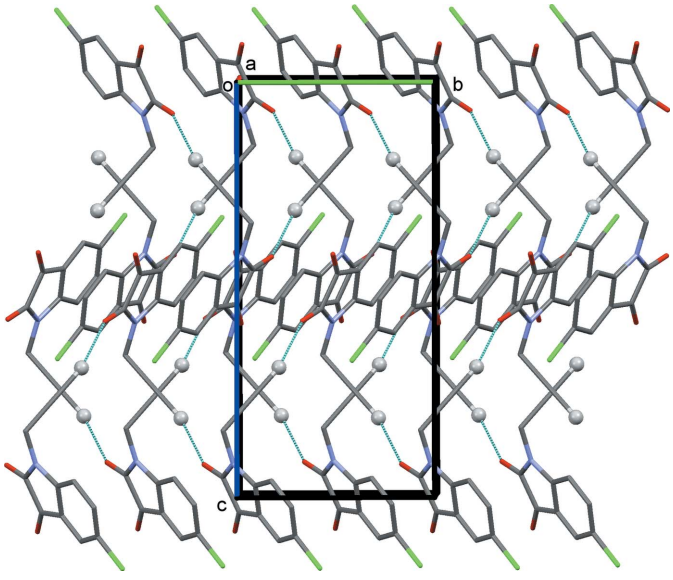


Figure 2
Crystal packing of the title compound, viewed along the *a* axis. The C—H···O hydrogen bonds are shown as dotted lines (see Table 1).

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.93	2.33	3.236 (3)	164

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Synthesis and crystallization

To a solution of 5-chloroindoline-2,3-dione (0.82 g, 5 mmol) in DMF (20 ml) potassium carbonate (1.04 g, 7.5 mmol) was added and the solution was stirred at room temperature. Propargyl bromide (0.7 ml, 7.5 mmol) was added dropwise and the resulting mixture was stirred overnight. After completion of the reaction (monitored by TLC), the mixture was partitioned between CH_2Cl_2 and water, and the CH_2Cl_2 layer was collected. The aqueous layer was extracted three times with CH_2Cl_2. The combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated under vacuum to obtain the desired product. Colourless block-like crystals were obtained by slow evaporation of a solution in chloroform.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_6ClNO_2$
M_r	219.62
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	15.0004 (4), 7.9035 (3), 16.8880 (6)
β (°)	101.057 (1)
V (Å^3)	1965.00 (12)
Z	8
Radiation type	Mo *K*α
μ (mm^{-1})	0.36
Crystal size (mm)	0.30 × 0.25 × 0.25
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.897, 0.913
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5667, 1723, 1429
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.092, 1.06
No. of reflections	1723
No. of parameters	136
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.25

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3* for Windows (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010).

Acknowledgements

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References

Bhrigu, B., Pathak, D., Siddiqui, N., Alam, M. S. & Ahsan, W. (2010). *Int. J. Pharm. Sci. Drug Res*, **2**, 229–235.
Bruker (2008). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Pandeya, S. N., Smitha, S., Jyoti, M. & Sridhar, S. K. (2005). *Acta Pharm.* **55**, 27–46.
Ramachandran, S. (2011). *Int. J. Res. Pharm. Chem.* **1**, 289–294.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Silva, J. F. M. da, Garden, S. J. & Pinto, A. C. (2001). *J. Braz. Chem. Soc.* **12**, 273–324.
Smitha, S., Pandeya, S. N., Stables, J. P. & Ganapathy, S. (2008). *Sci. Pharm.* **76**, 621–636.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

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5-Chloro-1-(prop-2-ynyl)indoline-2,3-dione

Crystal data

$C_{11}H_6ClNO_2$
M_r = 219.62
Monoclinic, $C2/c$
a = 15.0004 (4) Å
b = 7.9035 (3) Å
c = 16.8880 (6) Å
β = 101.057 (1)°
V = 1965.00 (12) Å^3
Z = 8
$F(000)$ = 896
D_x = 1.485 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1429 reflections
θ = 2.5–25.0°
μ = 0.36 mm^{-1}
T = 296 K
Block, colourless
0.30 × 0.25 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.897, T_{max} = 0.913
5667 measured reflections
1723 independent reflections
1429 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°, θ_{min} = 2.5°
h = −17→12
k = −9→7
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.092
S = 1.06
1723 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 1.521P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.62704 (12)	0.4097 (2)	0.47196 (10)	0.0358 (4)
C2	0.54523 (12)	0.3261 (3)	0.46003 (12)	0.0459 (5)
H2	0.5035	0.3370	0.4119	0.055*
C3	0.52684 (13)	0.2247 (3)	0.52229 (13)	0.0483 (5)
H3	0.4717	0.1676	0.5160	0.058*
C4	0.58942 (12)	0.2078 (2)	0.59317 (12)	0.0435 (5)
C5	0.67249 (12)	0.2900 (2)	0.60527 (11)	0.0390 (4)
H5	0.7146	0.2776	0.6531	0.047*
C6	0.69011 (11)	0.3908 (2)	0.54372 (10)	0.0342 (4)
C7	0.76974 (12)	0.4937 (2)	0.53727 (11)	0.0378 (4)
C8	0.74633 (13)	0.5761 (2)	0.45251 (11)	0.0409 (4)
C9	0.61323 (16)	0.5719 (3)	0.33937 (11)	0.0528 (5)
H9A	0.6327	0.6845	0.3275	0.063*
H9B	0.5486	0.5763	0.3391	0.063*
C10	0.62976 (14)	0.4554 (3)	0.27659 (11)	0.0505 (5)
C11	0.64529 (17)	0.3631 (4)	0.22730 (13)	0.0708 (7)
H11	0.6577	0.2894	0.1879	0.085*
Cl1	0.56412 (4)	0.08187 (8)	0.67021 (4)	0.0692 (2)
N1	0.66111 (11)	0.5215 (2)	0.41938 (8)	0.0420 (4)
O1	0.79434 (10)	0.67110 (19)	0.42323 (8)	0.0563 (4)
O2	0.84013 (9)	0.51612 (19)	0.58385 (8)	0.0544 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0398 (9)	0.0333 (10)	0.0348 (9)	0.0067 (8)	0.0081 (7)	−0.0063 (7)
C2	0.0385 (10)	0.0474 (12)	0.0479 (11)	0.0040 (9)	−0.0014 (8)	−0.0099 (9)
C3	0.0369 (10)	0.0430 (12)	0.0663 (13)	−0.0032 (9)	0.0135 (9)	−0.0109 (10)
C4	0.0454 (10)	0.0377 (11)	0.0530 (11)	0.0018 (8)	0.0238 (9)	−0.0007 (9)
C5	0.0403 (10)	0.0408 (11)	0.0367 (9)	0.0064 (8)	0.0096 (8)	−0.0012 (8)
C6	0.0341 (9)	0.0337 (10)	0.0358 (9)	0.0026 (7)	0.0090 (7)	−0.0049 (8)
C7	0.0381 (10)	0.0354 (10)	0.0408 (10)	0.0027 (8)	0.0094 (8)	−0.0056 (8)
C8	0.0504 (11)	0.0339 (10)	0.0424 (10)	0.0053 (9)	0.0186 (9)	−0.0031 (8)
C9	0.0693 (13)	0.0506 (13)	0.0364 (10)	0.0156 (11)	0.0051 (9)	0.0035 (9)
C10	0.0578 (12)	0.0583 (14)	0.0336 (10)	0.0107 (10)	0.0038 (9)	0.0031 (10)
C11	0.0820 (17)	0.0843 (18)	0.0424 (12)	0.0237 (14)	0.0027 (11)	−0.0117 (12)
Cl1	0.0749 (4)	0.0646 (4)	0.0789 (4)	−0.0021 (3)	0.0422 (3)	0.0151 (3)
N1	0.0526 (9)	0.0411 (9)	0.0317 (8)	0.0054 (8)	0.0067 (7)	0.0000 (7)
O1	0.0687 (9)	0.0482 (9)	0.0598 (9)	−0.0010 (7)	0.0316 (7)	0.0062 (7)
O2	0.0406 (8)	0.0610 (10)	0.0586 (9)	−0.0075 (7)	0.0016 (7)	−0.0006 (7)

Geometric parameters (Å, º)

C1—C2	1.374 (3)	C6—C7	1.466 (2)
C1—C6	1.395 (2)	C7—O2	1.203 (2)

C1—N1	1.416 (2)	C7—C8	1.550 (3)
C2—C3	1.391 (3)	C8—O1	1.210 (2)
C2—H2	0.9300	C8—N1	1.363 (2)
C3—C4	1.379 (3)	C9—N1	1.459 (2)
C3—H3	0.9300	C9—C10	1.461 (3)
C4—C5	1.385 (3)	C9—H9A	0.9700
C4—Cl1	1.7367 (19)	C9—H9B	0.9700
C5—C6	1.375 (2)	C10—C11	1.163 (3)
C5—H5	0.9300	C11—H11	0.9300

C2—C1—C6	120.91 (17)	O2—C7—C6	131.19 (18)
C2—C1—N1	128.89 (17)	O2—C7—C8	123.86 (17)
C6—C1—N1	110.20 (15)	C6—C7—C8	104.95 (15)
C1—C2—C3	117.85 (17)	O1—C8—N1	127.83 (18)
C1—C2—H2	121.1	O1—C8—C7	126.21 (18)
C3—C2—H2	121.1	N1—C8—C7	105.95 (15)
C4—C3—C2	120.75 (18)	N1—C9—C10	112.24 (16)
C4—C3—H3	119.6	N1—C9—H9A	109.2
C2—C3—H3	119.6	C10—C9—H9A	109.2
C3—C4—C5	121.73 (18)	N1—C9—H9B	109.2
C3—C4—Cl1	119.72 (15)	C10—C9—H9B	109.2
C5—C4—Cl1	118.55 (15)	H9A—C9—H9B	107.9
C6—C5—C4	117.27 (17)	C11—C10—C9	178.3 (2)
C6—C5—H5	121.4	C10—C11—H11	180.0
C4—C5—H5	121.4	C8—N1—C1	111.27 (15)
C5—C6—C1	121.47 (16)	C8—N1—C9	123.39 (17)
C5—C6—C7	130.92 (16)	C1—N1—C9	125.34 (17)
C1—C6—C7	107.61 (15)		

C6—C1—C2—C3	1.1 (3)	C1—C6—C7—C8	−0.14 (18)
N1—C1—C2—C3	−178.77 (17)	O2—C7—C8—O1	−0.2 (3)
C1—C2—C3—C4	−0.7 (3)	C6—C7—C8—O1	180.00 (17)
C2—C3—C4—C5	−0.1 (3)	O2—C7—C8—N1	−179.26 (17)
C2—C3—C4—Cl1	179.38 (14)	C6—C7—C8—N1	0.98 (18)
C3—C4—C5—C6	0.4 (3)	O1—C8—N1—C1	179.53 (18)
Cl1—C4—C5—C6	−179.05 (13)	C7—C8—N1—C1	−1.47 (19)
C4—C5—C6—C1	0.0 (3)	O1—C8—N1—C9	0.4 (3)
C4—C5—C6—C7	179.80 (17)	C7—C8—N1—C9	179.41 (15)
C2—C1—C6—C5	−0.8 (3)	C2—C1—N1—C8	−178.69 (18)
N1—C1—C6—C5	179.11 (15)	C6—C1—N1—C8	1.5 (2)
C2—C1—C6—C7	179.39 (16)	C2—C1—N1—C9	0.4 (3)
N1—C1—C6—C7	−0.73 (19)	C6—C1—N1—C9	−179.44 (16)
C5—C6—C7—O2	0.3 (3)	C10—C9—N1—C8	90.9 (2)
C1—C6—C7—O2	−179.88 (19)	C10—C9—N1—C1	−88.1 (2)
C5—C6—C7—C8	−179.97 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.93	2.33	3.236 (3)	164

Symmetry code: (i) −*x*+3/2, *y*−1/2, −*z*+1/2.